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Development of a refractory insulating brick

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DEVELOPMENT OF A REFRACTORY INSULATING BRICK

BY

EDWIN L. DUDLEY

A

THESIS

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Approved By

Associate Professor of Ceramic Department.

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Development of A Refractory Insulating Brick

INTRODUCTION

The demand for a greater capacity and higher efficiency, particularly during the past decade or two, has resulted in the use of much higher temperatures in industrial processes. Relatively small increases in the operating temperature often mean considerable increases in the capacity of the equipment. For example, the steaming capacity of a boiler, the melting time in a metallurgical furnace and the output of a portland cement kiln are all influenced greatly by the amount of heat supplied in the operation.

High temperature insulation, while a comparatively recent development, has played an important part in this stepping up of industrial temperatures. Heat flow through refractory walls of furnaces and similar equipment carries a large percentage of the heat to the outside surface, from which it is lost by radiation and convection. With higher temperatures heat losses are greatly increased unless the furnace inclosure is designed to provide added resistance to heat flow. For the successful operation of such equipment heat losses must be minimized by the use of adequate insulation; first, to reduce costs by lowering the quantity of heat necessary (to supply) to bring about the desired result; second, to make possible accurate temperature control;

and third, to insure more uniform heat distribution within the equipment.

In the loss of heat from a furnace wall, there is involved, first the transmission by conduction through the wall to the outer surface; and second, the giving out of the heat from the outer surface to the air by radiation, enhanced by convection due to air movement of the wall. No more heat can be transmitted through the wall, at equilibrium, than is emitted by the outer surface of the wall.

The quantity of heat transmitted by conduction through the wall varies directly with the area of the wall, the temperature difference between the hot and cold surfaces of the wall, the thermal conductivity of the materials of which the wall is composed, and inversely with the thickness of the wall.

(1)

The rate of emission of heat from the cooler face of the wall depends upon the various factors --- mainly, upon the surface temperature, the temperature difference between the wall and the adjacent air, the rate of circulation of air, and, in lesser degree upon the nature of the surface of the wall, its color, position, degree of smoothness, and the material of construction. Radiation losses rise rapidly with rise in temperature, and is consequently important to reduce the heat loss at the higher temperature.

The following statement shows the position among the furnace

construction materials which the new refractory brick will probably occupy.

(2)
Mr. Pike makes the suggestion that research be followed along the lines of experimentation with the various sizes and numbers of pore spaces in a given volume, combined with various refractory bodies.

For many years there was a general belief that since air was a poor conductor of heat, air spaces built in the walls of boilers, furnaces, etc., would prevent or reduce losses of heat through the walls. Messrs. Ray and Kreisinger of the U. S. Bureau of Mines (3) ran a series of exhaustive tests on air spaces as used in insulation and shows that this artifice defeats its own purpose at high temperature because of the fact that the hot surfaces of the air space radiate heat so rapidly that a greater amount is lost across a space by radiation than would be conducted were the air spaces filled with material of relative high conductivity such as fire brick. The reason for this is easily seen when we realize the quantity of heat passing through a portion of solid wall by conduction depends on the difference between the temperature of two planes limiting the portion of the wall; while the quantity of heat that passes air space on a wall depends on the difference between the fourth powers of the absolute temperature; of the surface enclosing the air space. The attempted insulation of furnace walls by the

use of so-called "dead air space" is thus confirmed fallacious.

(4)
A good refractory material is essentially strong and dense and is consequently highly conductive. In order to prevent excessive heat loss, a furnace wall must either be built up of great thickness, which is costly, give an inelastic wall and absorb a large amount of heat not used in productive work, or be built to include as component of the wall, a layer of some material of lower thermal conductivity, called the insulator.

(5)
The rate of heat flow through a wall depends upon the resistance of the component materials to passage of heat. Generally speaking, a material of low apparent density is considered to be a good insulator. Most of such materials have a number of small "voids" or cells containing air. It is generally thought that the effectiveness of such products is due principally to the insulating properties of the air spaces themselves. Actually, however, the low conductivity of these materials is due primarily to the great number of surfaces imposed in the path of heat flow, each one of which offers an appreciable resistance to the emission of heat.

Percent porosity, therefore, may not be a true index to conductivity. To be effective as an insulation, particularly at high temperatures, a material must not only have a high porosity but the individual pores must be microscopic in size to provide a maximum number of surfaces from which heat must radiate.

There are many materials which are suitable for insulation

of lower temperature ranges. But the most common methods used in commercial work are: I. By any combustible materials to the raw clay and then burning out the material leaving small air holes in its place. II. By bloating the brick while it is in the plastic stage by means of a chemical action within the clay mass which will generate small bubbles of gas and cause the mass to swell in much the same way as yeast or baking powder causes a soft mass of dough to rise.

Some manufacturers are grinding coal or coke with the shale or clay in the dry pan. One or two plants mine coal with their clay in whatever portions these materials happen to occur, while other manufacturers add a given amount of combustible to the clay at the dry pan. This is a very cheap operation, but makes the production of uniform ware very difficult.

In the high temperature field, the insulating materials must not only have a low conductivity but must also have a high degree of refractoriness, a quality not ordinarily found in insulating material. The addition of coal, coke, cork, or other materials produce fairly high porosity but the refractoriness of the insulator is usually lowered by the fluxing of the ash left after firing. Organic material such as naphthalene has been successfully substituted for some of the other organic material in this field.

PURPOSE

The method of bloating brick of low refractoriness has been accomplished by the addition of carbonates of the alkalines and alkaline earths which when acid is applied give off CO_2 to form a porous structure. It was thought that if a material which would not act as a flux might even enhance the refractoriness of the clay used a high temperature insulator might be produced which would compare favorably to those now produced by the use of volatiles.

An aluminum salt which when combines with a non-metallic base that would generate a gas was selected. The aluminum in the process of firing would oxidize to the refractory Al_2O_3 and the base would volatilize without detriment to the refractoriness of the brick.

PROCEDURE I

Materials used:

The materials used in the study were: 1. Empire Fire clay obtained from the A. P. Green Firebrick Company at Mexico, Missouri. 2. Cheltenham Fire Clay. 3. Chemically pure ammonium carbonate, $(\text{NH}_4)_2\text{CO}_3$. 4. Chemically pure aluminum Chloride, Al Cl_3 .

Method of procedure:

In the first series of experiments the clay used was ground to pass through a twenty-mesh screen on a forty-mesh, the fines being discarded. The aluminum chloride and the ammonium carbonate were found to combine in the ratio of twenty six parts of aluminum chloride to twenty eight parts of ammonium carbonate. A series was made up according to Table I.

TABLE I

| Sample No. 3000 gm Sples. | $(\text{NH}_4)_2\text{CO}_3$ | Al Cl_3 | Addition % of Clay Batch |
|------------------------------|------------------------------|------------------|-----------------------------|
| | 28 Parts | 26 Parts | |
| I | 3.9 gm/M | 3.6 gm/M | .25 |
| II | 7.8 " | 7.2 " | .56 |
| III | 11.7 " | 10.8 " | .75 |
| IV | 15.6 " | 14.4 " | .100 |
| V | 19.5 " | 18.0 " | 1.50 |
| VI | 23.4 " | 21.6 " | 2.00 |
| VII | 27.3 " | 25.2 " | 2.50 |

In each case the aluminum chloride was finely ground and mixed with the clay mass until it was thoroughly homogenous; whereas, the ammonium carbonate was dissolved in approximately 500cc of tempering water. The body was worked to permit the ammonium carbonate in the water to come in contact with the entire mass. and to bring it to the right consistency . It was then poured into small four-sided wooden molds, $4\frac{1}{2}$ X 9" X 5". The mold was greased and set on a metal plate that was to serve as a bottom. The samples were placed in an electric dryer and were dried at approximately 200° C. for 12 hours. At the end of that period they were taken out and inspected. It was found that each specimen showed a considerable increase in volume. The dried samples were then fired in an oil-fired, high temperature kiln, according to the following chart Fig. I to cone 12.

The fired specimens were cut into small blocks and weighed dry and then boiled in vaseline for an hour. The vaseline was allowed to harden and then the blocks were removed from the pan of vaseline, and the excess vaseline was removed from the sides of the blocks. The vaseline-saturated blocks were again weighed, and also weighed suspended in water. The vaseline filled all the open pores. The specific gravity of the vaseline was obtained by weighing in a pycnometer. The specific gravity was found to be .835 a sample of each specimen was obtained by chipping off the corners of the fired block, and grinding in a mortar of 160 mesh.

The true specific gravity of the clay body was then obtained by weighing the powdered body in a pycnometer according to the standard method for obtaining true specific gravities of powdered substances.

Formula used:

Data on true specific gravity of a fired body:

| | |
|------------------------------------|---------|
| Wt. of empty pycnometer = | 18.2138 |
| Wt. of pycnometer / water = | 68.1392 |
| Wt. of pycnometer / clay = | 28.2314 |
| Wt. of pycnometer / sample / water | 73.4921 |

$$\text{True } S_p. G. = \frac{28.2314 - 18.2138}{(28.2314 - 18.2138) - (73.4921 - 68.1392)}$$

$$= \frac{10.0176}{10.0176 - 5.3529}$$

$$\frac{10.0176}{4.6647} = 2.1475$$

With the weights and specific gravities obtained, the volumes and percenta of open, closed and total pores were obtained.

The calculations were made in the followinf manner:

$$\text{Bulk Vol.} = \text{Wt. in vaseline} - \text{Wt. Suspended in } H_2O$$

$$\text{Open pore Vol.} = \frac{\text{Wt. Soaked in vaseline} - \text{dry Wt.}}{\text{Specific gravity of vaseline}}$$

$$\% \text{ Open pore Vol.} = \frac{\text{Open pore Vol.}}{\text{Bulk Vol.}} \times 100$$

True Vol. = Bulk Vol. - Open pore Vol.

% Porosity = Bulk Vol. $\frac{\text{Wt. Dry}}{\text{True Specific Gravity of body} \times 100}$
Bulk Vol.

% Closed pores = % Total pores - % open pores

The porosity obtained from the blocks made in Procedure I
are shown in the following tables.

PROCEDURE II

In this procedure both the ammonium carbonate and the aluminum chloride were finely ground in the dry state and added to the clay mass together. The mixture was thoroughly difused throughout the clay mass, and the tempering water was then added. IN this procedure the same amount of tempering water was added, and the conditions were approximately the same as in Procedure I. The porosities obtained are given in Table Iv of Procedure II.

PROCEDURE III

In this procedure both Empire fire clay and Cheltenham were used. The clay was ground to pass a sixty-mesh screen, and the fines were discarded. The aluminum chloride and ammonium carbonate was finely ground in a mortar before being added to the clay. After being thoroughly mixed, the body was run through a forty-mesh screen several times. This was to make the mixture thoroughly homogenous, before the tempering water was added. The amount of water used in the experiment was considerably less than that used in the previous procedures. Just enough was added to make a stiff, pasty mass, and this was immediately placed in a dryer at 250° C. The porosities obtained are shown in Table V.

DISCUSSION OF DATA

Procedure I:

In procedure I the aluminum chloride was ground in the clay mass and the ammonium carbonate was dissolved in the tempering water. Upon the addition of the water there was a violent reaction that took place and it was very difficult to control the gas generated. The total percent porosity increased steadily, however, up until 2.0 percent of the mixture was added. Above 2.0 percent the reaction was so violent that most of the gas escaped and the porosity decreased. The percentage of closed pores proved to be very irregular, sample No. II and sample No. VII were the only two which showed the least signs of regularity. This was no doubt due to the methods of procedure, and unlike conditions resulting.

Procedure II:

In procedure II the aluminum chloride and the ammonium carbonate were ground together dry and the tempering water was added. Much better results were obtained, but there was no uniformity of results. An inspection of the data obtained shows that Sample I containing .25 of one percent of the mix showed the highest total percent porosity, while sample VI containing 2.0 percent showed a 13.0 percentage of closed pores and 57.7 percentage of total porosity. This indicated that conditions were not constant, that perhaps the same degree of plasticity

was not reached, or there was a variation in grain size, or rate of pouring into mold.

Procedure III:

The aluminum chloride and ammonium carbonate were ground dry in the clay to pass a sixty-mesh screen, was carefully mixed and just enough tempering water was added to form a real stiff mass. The best results by far were obtained by this method. The highest percent of closed pores was obtained by sample III containing .75 of one percent of the mix. As before, the results were far from uniform, but as a whole this method produced a much higher percentage of the desired closed pores.

TABLE III

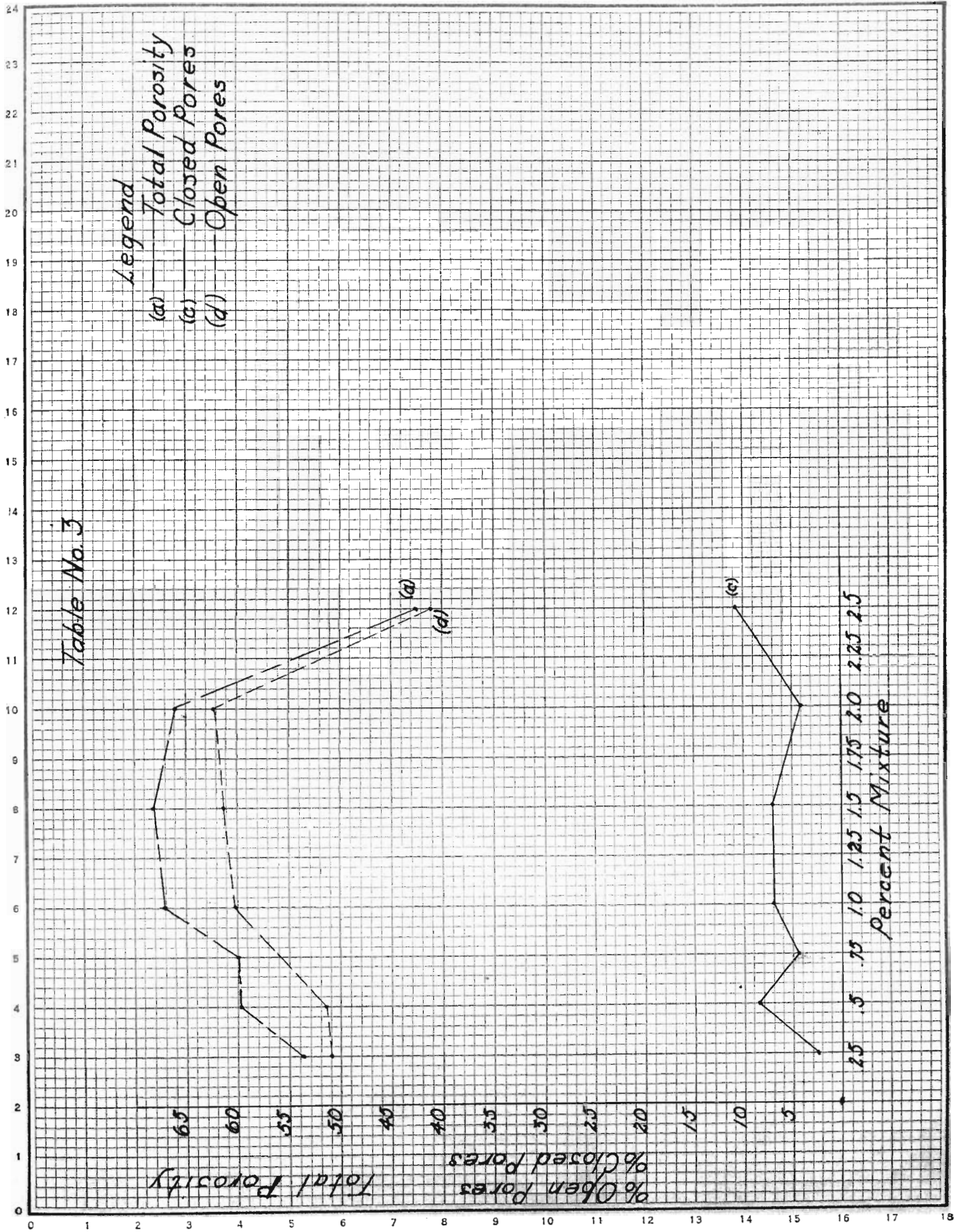
PROCEDURE ONE

SUMMARIZED DATA

| SAMPLE | DRY WT. | WT. SOAKED VASELINE | WT. SAT. SUSP. H ₂ O | BULK VOL. | TRUE VOL. | OPEN PORES VOL. | % OPEN PORE VOL. | %CLOSED PORES | % TOTAL PORE VOL. |
|--------|---------|------------------------|------------------------------------|--------------|--------------|-----------------------|------------------------|------------------|-------------------------|
| I | 219.50 | 291.20 | 123.20 | 168 | 82.14 | 81.86 | 51.11 | 2.74 | 53.9 |
| II | 145.62 | 211.91 | 78.00 | 133.91 | 54.53 | 79.38 | 51.37 | 8.56 | 59.93 |
| III | 164.20 | 236.8 | 87.20 | 149.60 | 62.66 | 86.94 | 55.55 | 4.46 | 60.01 |
| IV | 147.00 | 220.90 | 77.80 | 143.10 | 54.60 | 88.50 | 60.19 | 7.08 | 67.20 |
| V | 157.30 | 235.30 | 82.50 | 153.45 | 59.69 | 93.76 | 61.10 | 7.01 | 68.11 |
| VI | 167.25 | 266.85 | 87.70 | 179.15 | 117.04 | 111.28 | 62.11 | 4.22 | 66.33 |
| VII | 75.02 | 100.06 | 28.90 | 71.16 | | 29.3 | 41.02 | 10.76 | 42.4 |

Table No. 3

Legend
 (a) --- Total Porosity
 (a) --- Closed Pores
 (d) --- Open Pores



| SAMPLE | DRY WT. | WT.SOAKED VASELINE | WT. SAT. SUSP.H ₂ O | BULK VOL. | TRUE VOL. | OPEN PORE | % OPEN PORE | % CLOSED PORE | % TOTAL PORE |
|--------|---------|-----------------------|-----------------------------------|--------------|--------------|--------------|----------------|------------------|-----------------|
| I | 75.18 | 112.99 | 28.20 | 84.09 | | 44.30 | 52.60 | 10.2 | 62.8 |
| II | 70.45 | 104.33 | 26.88 | 77.45 | | 38.5 | 49.7 | 12.6 | 62.3 |
| III | 62.82 | 94.40 | 22.53 | 71.87 | | 37.6 | 51.4 | 12.3 | 63.7 |
| IV | 71.96 | 104.24 | 27.79 | 76.76 | | 37.89 | 49.1 | 11.9 | 56.1 |
| V | 74.35 | 100.85 | 30.43 | 70.42 | | 31.1 | 44.2 | 11.9 | 56.1 |
| VI | 73.23 | 102.39 | 27.79 | 74.6 | | 34.2 | 45.8 | 13.9 | 57.7 |
| VII | 75.30 | 90.04 | 35.90 | 54.14 | | 17.2 | 31.8 | 10.6 | 42.4 |

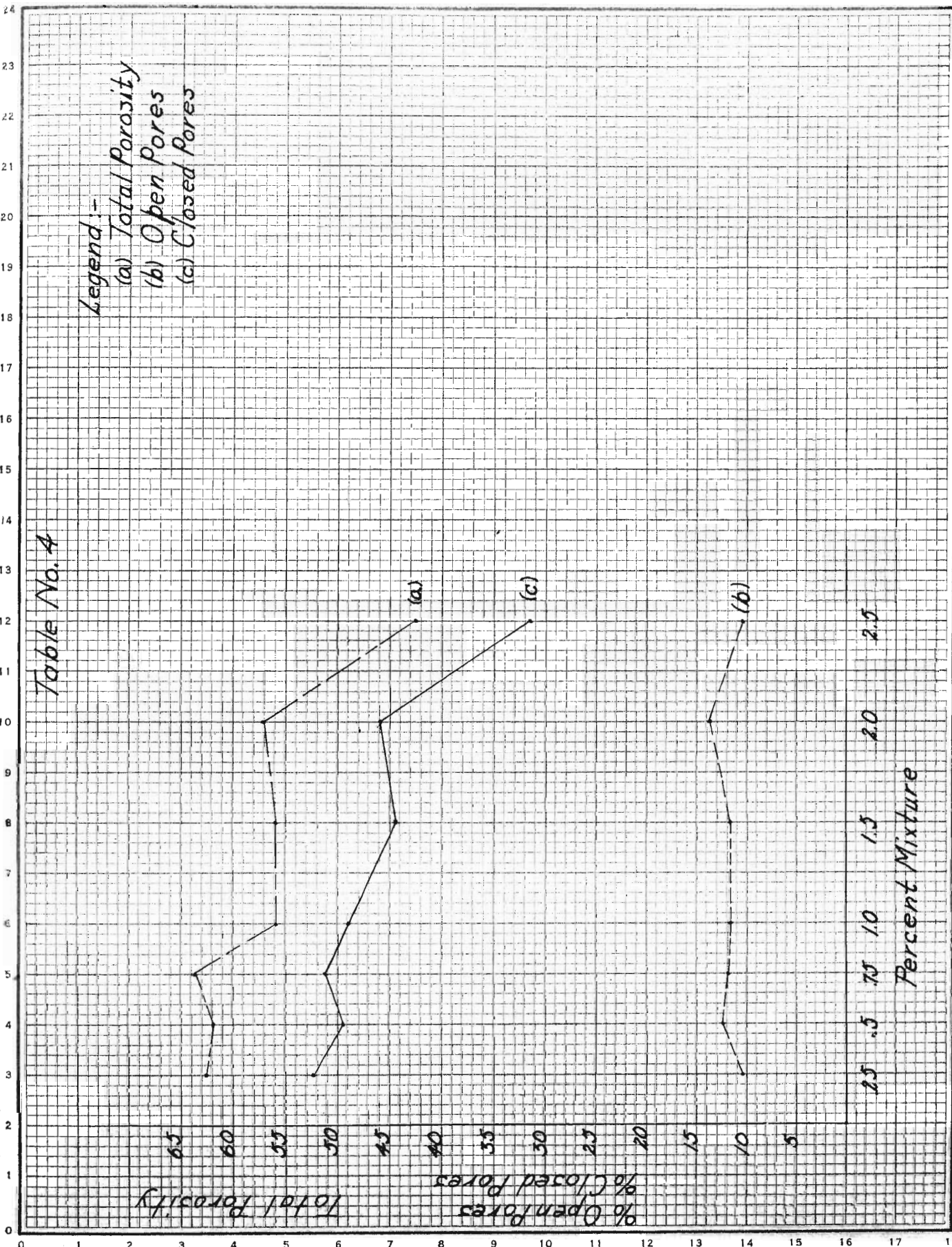
Summarized Data

Procedure II

TABLE IV

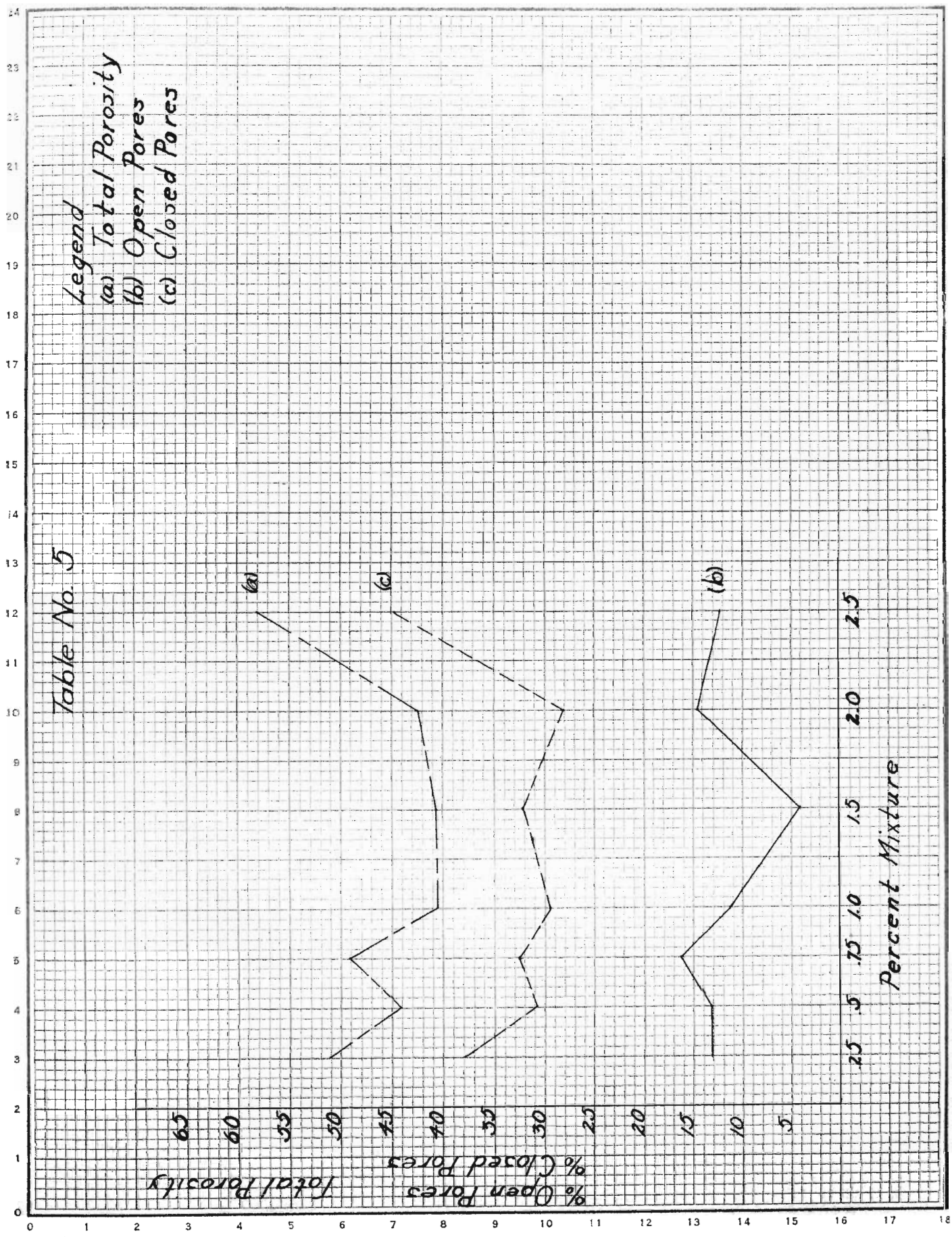
Table No. 4

Legend:-
 (a) Total Porosity
 (b) Open Pores
 (c) Closed Pores



| SAMPLE | WT. DRY | WT.SOAKED VASELINE | WT. SAT. SUSP. H_2O | BULK VOL. | OPEN PORE VOL. | % OPEN PORE VOL. | % CLOSED PORES | % TOTAL PORES |
|--------|---------|-----------------------|--|--------------|----------------------|------------------------|-------------------|------------------|
| I | 78.64 | 100.21 | 33.66 | 66.55 | 25.3 | 38.0 | 13.0 | 51.0 |
| II | 75.35 | 90.12 | 34.30 | 55.82 | 17.3 | 30.9 | 13.1 | 44.0 |
| III | 108.22 | 133 .09 | 44.00 | 89.09 | 29.0 | 32.5 | 16.1 | 49.4 |
| IV | 52.28 | 61. 36 | 24.93 | 36.43 | 10.7 | 29.4 | 11.1 | 40.5 |
| V | 57.94 | 69.08 | 28.32 | 40.86 | 13.1 | 32.1 | 4.0 | 41.1 |
| VI | 47.72 | 55.94 | 21.56 | 34.38 | 9.64 | 28.0 | 14.4 | 42.4 |
| VII | 53.29 | 71.57 | 23.78 | 49.79 | 21.4 | 44.8 | 12.2 | 58 |

TABLE V
Procedure III
Summarized Data



SUMMARY

In the preceding experiments it was found that when the tempering water was added to the clay mass containing the aluminum chloride and the ammonium carbonate, a violent reaction took place. This reaction liberated the Co_2 necessary to produce the desired porous structure of the clay body, and much of the gas escaped before it could even be thoroughly mixed with the clay. Several attempts were made to overcome this difficulty, but with little success. The first method tried was grinding the aluminum chloride in the clay and adding the ammonium carbonate in the tempering water. This was found to be unsatisfactory and slightly better results were found by grinding both the aluminum chloride and the ammonium carbonate dry with the clay and then adding the tempering water.. The best results were obtained, however, by grinding the clay, aluminum chloride and the ammonium carbonate to pass a sixty-mesh screen. To this finely-ground body just enough tempering water was added to make a very stiff mud mix. This was immediately placed in an electric dryer at 250°C .

An inspection of the results obtained by the various methods of procedure showed that the number of closed pores increased as the size of the grains decreased, and that both the number of closed pores and open pores increased as the plastic consistency was increased.

Much more reliable data could have been obtained in this experiment had it been possible to obtain the same grain size, and the same plastic consistency each time. (These experiments showed, however, that the factor to which porosity is most sensitive is the consistency to which the plastic clay mass is made up before it is placed in the molds. The right consistency seems to be the point where the clay mass is barely soft enough to flow).

CONCLUSIONS

1. That an insulating brick can be made using aluminum chloride and ammonium carbonate to impart a porous structure to the brick.
2. That the degree of porosity depends upon the degree of plasticity, fineness of grain size, rate of drying, and percentage of mixture used.
3. That the cost of manufacture would be such as to make the brick practical and profitable.

APPLICATION TO INDUSTRY

The indications are that a soft mud process could be used in the manufacture of an insulating brick using Part III of procedure III. The addition of aluminum chloride and ammonium carbonate could be made to the clay in the dry pan, provided the clay is dry. The grinding and subsequent screening would have a tendency to distribute throughout the mix. The tempering water could be added in a pug mill and the soft mass extruded from an auger machine into molds constructed of metal. These molds would be fitted with a metal top so that when the top had been raised to a certain point it would strike a lug which would arrest its movement upward.

The further expansion of the gas bubbles would cause them to elongate which shape might be advantageous to an insulator. The mold would thus give property of definite size and shape without the necessity of regrinding.

The molds of metal could be placed directly in the drier which would be necessary to the generation of the gas.

The brick could be set at top of a kiln of regular brick in order to relieve the load which would be superimposed in a kiln set with insulators alone.

The cost of such brick would be comparatively low, considering that aluminum chloride and ammonium carbonate can both be

obtained for from six to eight cents per pound in wholesale lots.

RECOMMENDATIONS FOR FUTHER RESEARCH

For furthur research I would suggest grinding the clay and aluminum chloride with the ammonium carbonate together to pass a eighty or one-hundred mesh screen, and adding just enough tempering water to make a very stiff soft mud mass. This could be placed into a kiln or very high temperature dryer. It is believed that by this method a brick of very high porosity can be economically produced.

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E.L.D.